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ORIGINAL

# Comparison of *Pinus taeda* L. whole-tree wood property calibrations using diffuse reflectance near infrared spectra obtained using a variety of sampling options

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**Abstract** A necessary objective for tree-breeding programs, with a focus on wood quality, is the measurement of wood properties on a whole-tree basis, however, the time and cost involved limits the numbers of trees sampled. Near infrared (NIR) spectroscopy provides an alternative and recently, it has been demonstrated that calibrations based on milled increment cores and whole-tree data can provide good estimates of whole-tree properties. Several options exist for sampling standing trees and the aim of this study was to compare wood property calibrations based on NIR spectra collected from samples obtained using different sampling methods. Calibrations for whole-tree lignin and basic specific gravity based on NIR spectra from whole-tree chips (milled or intact) had the strongest statistics, calibrations based on NIR spectra from milled increment cores were similar. Other options for sampling the tree (drill shavings, etc.) gave errors that were too large for practical applications. If an increment core is going to be used to estimate whole-tree properties, it is recommended that it be dried and milled prior to analysis.

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## Introduction

A necessary objective for any tree-breeding program, with a focus on wood quality, is the measurement of whole-tree properties. Measurement of whole-tree properties generally requires trees to be destructively sampled with bolts or discs taken to provide a composite sample that represents the whole-tree. The composite samples are analyzed and depending on the property of interest, several standard methods may be utilized. Destructive sampling is extremely time-consuming and owing to practical constraints, the number of trees that can be sampled is limited. An option to destructive sampling for the purpose of obtaining a whole-tree composite is to take an increment core from a standing tree, grind it and use the milled wood for all chemical analysis and assume that the results are indicative of the whole-tree.

While the difficulties of destructive sampling limit, the number of trees that can be sampled, the costs associated with chemical analysis also limits the number of samples that can be analyzed, particularly if the property is expensive and difficult to measure. Spectroscopic methods, such as mid infrared (MIR) and near infrared (NIR) spectroscopy, provide a rapid alternative to traditional methods of wood property assessment. NIR spectroscopy has been widely used for the estimation of several wood properties including pulp yield, cellulose and lignin content on a whole-tree basis (Birkett and Gambino 1988; Michell 1995; Michell and Schimleck 1998; Olsson et al. 1995; Schimleck et al. 2000; Wright et al. 1990) The method involves collecting the NIR spectra of a set of characterized samples, developing a regression equation [commonly using partial least squares (PLS) regression] and using the equation to predict the wood properties of further samples (the validation set) based on their NIR spectra. The spectra are typically collected from milled wood chips (representing a whole-tree, or even several trees) and consist of overtone and combination bands of the fundamental stretching vibrations of O–H, N–H and C–H functional groups (Osborne et al. 1993). To obtain nondestructive estimates of whole-tree properties, calibrations must be applicable to samples that can be obtained from standing trees nondestructively, such as increment cores.

If whole-tree wood properties are going to be estimated using increment core NIR spectra, calibrations must be developed between whole-tree properties (obtained from destructively sampled trees) and NIR spectra collected from increment cores. In a study based on several eucalypt species and hybrids grown in Brazil, Schimleck et al. (2006) demonstrated that calibrations for several properties, including basic density, lignin content and pentosans, based on whole-tree data and NIR spectra collected from increment cores (taken from 0.65 and 1.50 m) provided similar calibration statistics to those obtained using whole-tree composite chips. When the calibrations (based on increment core and composite chip NIR spectra) were applied to a separate test set the increment core and composite chip calibrations performed in a similar manner indicating that the estimation of whole-tree properties using calibrations based on NIR spectra obtained from increment cores was possible. In studies of hybrid poplar (Schimleck et al. 2005b) and *Eucalyptus nitens* (Deane and Maiden) Maiden (shining gum) (Schimleck et al. 2005a) calibrations based on whole-tree chip and increment core NIR spectra, also provided similar calibration statistics.

These studies have been based on hardwoods, specifically eucalypts and hybrid poplar, and the utilization of core NIR spectra for the estimation of whole-tree properties has not been investigated for softwoods. In the Southern USA, the most commercially important softwood is *Pinus taeda* L. (loblolly pine). While its wood properties are inferior to longleaf pine (*Pinus palustris*), it has been widely planted in the Southern USA because of its ability to grow well on a wide variety of sites, and increasingly solid wood and fiber production in the Southern USA is from plantation grown *P. taeda*. Owing to its importance as a plantation species, *P. taeda* has been subjected to genetic improvement with an increasing emphasis on improving wood properties (Li et al. 1999). Hence the nondestructive evaluation of *P. taeda* wood properties is an important objective.

While collecting increment cores is the most common method for sampling standing trees several variations on taking cores exist, for example collecting drill shavings, collecting NIR spectra directly from the surface of a hole drilled into a tree (So et al. 2004), etc., and it is the aim of this study to examine wood property calibrations obtained using NIR spectra collected from samples obtained using a variety of different sampling options. Our specific objectives are to:

1. Sample selected *P. taeda* trees using a variety of different sampling methods and collect NIR spectra from the various samples [wood properties (basic specific gravity, chemical composition) of the trees, based on whole-tree composite chips, will be measured using standard analytical techniques].
2. Develop wood property calibrations for the measured traits using PLS regression and NIR spectra collected from the different wood samples.
3. Compare calibrations to determine what impact the different methods used to collect NIR spectra have on calibration error and recommended suitable approaches for the non-destructive estimation of whole-tree properties using NIR spectroscopy.

These objectives help answering the hypotheses that NIR can be used to rapidly determine whole-tree wood properties in *P. taeda* and accuracy of calibrations can be increased by using different representative samples.

## Materials and methods

### Sampling selected trees

Fourty *P. taeda* trees from a half-sib progeny trial planted at International Paper's Southlands facility were selected for sampling. The trees were selected from a total of 106 trees aged 13 years. Height and diameter at breast height had been measured periodically, and increment cores removed at breast height were used for the measurement of basic specific gravity, cellulose and lignin content (cellulose and lignin content were both estimated by International Paper using NIR spectroscopy). The Fourty trees were selected to encompass the range of the measured properties. Prior to sampling, selected trees were checked for fusiform rust and pitch canker, and care was taken to avoid sampling any trees with noticeable cankers. The

selected trees were felled and samples removed for wood property analysis and NIR analysis. The following samples were removed:

- Four bolts (0.75 m long) per tree with each bolt cut in the center of each 25% of total height.
- Disks (25 mm thick) at 1.5 m intervals up the stem of each tree.
- Two 12 mm increment cores from each tree at breast height.
- One 0.3 m length bolt from breast height (for later NIR analysis).
- Shavings from drilling a 6.25 mm hole into the tree at breast height.

## Sample preparation and analysis

The Institute of Paper Science and Technology's (IPST) chipper was used to chip the four 0.75 m bolts. The chips were thoroughly mixed to give a composite sample. A subsample was removed for NIR analysis and for the determination of wood sugars and lignin content by IPST staff. The disks cut at each height level were used to determine weighted whole-tree basic specific gravity (Disc BSG) using standard methods. International Paper staff determined whole-tree specific gravity using the composite chips (Chip BSG).

IPST staff was provided with a subsample of each composite, which was used for the determination of the chemical constituents of wood. Cellulose, total sugars and individual sugars (Arabinan, Galactan, Glucan, Mannan and Xylan) were determined using acid hydrolysis and high performance anion exchange chromatography with pulsed amperometric detection (HPAEC-PAD). Klason lignin and acid soluble lignin were determined using standard methods. The calibrations for wood sugars were poor and, subsequently, are not reported in this study. A summary of the insoluble lignin, Chip BSG and Disc BSG data are given in Table 1.

## Collection of NIR spectra from selected trees

NIR spectra were collected from a variety of different samples, a description of each follows.

**Table 1** Statistical summary of each property examined (Chip BSG, Disc BSG and insoluble lignin)

| Wood property | Maximum | Minimum | Mean | Std. dev. |
|---|---|---|---|---|
| Chip BSG | 0.55 | 0.39 | 0.46 | 0.04 |
| Disc BSG | 0.56 | 0.41 | 0.47 | 0.04 |
| Insoluble lignin | 32.25 | 28.52 | 29.97 | 0.82 |

*Chip BSG* chip basic specific gravity, *Disc BSG* disc basic specific gravity

*NIR spectra from whole-tree wood chips*

NIR spectra over the wavelength range 400–2,500 nm were collected from each whole-tree chip sample while green using a FOSS NIRSystems 6500 (wavelength range 400–2,500 nm) fitted with a large transport module. The chips were dried at 50°C for 24 h (all subsequent material that is referred to as dried was also dried using this method) and a second set of NIR spectra collected using the same instrument. The chips were then milled and NIR spectra of the ground samples were collected using a FOSS NIRSystems 5000 (wavelength range 1,100–2,500 nm) fitted with a spinning sample module.

*NIR spectra from core holes*

An attempt was made to collect NIR spectra from one of the holes created when the breast height core was taken using an Ocean Optics fiber-optic probe system (wavelength range 856–1,736 nm). Owing to difficulties with measurement (the fit of the probe into the hole was tight and resin accumulated on the probe), the 0.3 m bolt cut at breast height was used for NIR measurements in our laboratory with a slightly larger hole drilled by an auger bit. NIR spectra were collected near the bark, the pith and at a point midway between the two. The three spectra were averaged to give a single spectrum per tree for calibration development.

*NIR spectra from core samples*

For one of the breast height cores NIR spectra were collected from the rough surface of the cores while green and dry and from a smooth surface (cut with a bandsaw) also when green and dry. Spectra were collected in 10 mm increments from the radial surface using a FOSS NIRSystems 5000 and in 5 mm increments using an Ocean Optics fiber-optic probe system. All measurements were made from pith to bark.

The second core from each tree was dried and milled in a Wiley number 4 mill fitted with a 1 mm screen. NIR spectra were collected from each of the milled cores using a FOSS NIRSystems 5000 fitted with a spinning sample module.

*NIR spectra from drill shavings*

For each selected tree, shavings were collected by drilling a 6.25-mm hole at breast height. NIR spectra were collected from the shavings when green, dried, and dried and milled using a FOSS NIRSystems 5000 fitted with a spinning sample module.

## Development of calibrations for Chip BSG, Disc BSG and lignin

Calibrations for Chip BSG, Disc BSG and lignin were developed using partial least squares (PLS) regression with full cross validation and a maximum of 10 factors

(Unscrambler, version 8.0, Camo AS, Norway). Calibrations were developed using second derivative spectra, left and right gap widths of 8 nm were used for the conversion. For the FOSS instruments this limited the wavelength range available for calibration development to 1,108–2,492 nm (wavelengths in the range 400–1,100 nm collected by the FOSS 6500 were not used). The wavelength range utilized for the Ocean Optics instrument was 870–1,710 nm. Depending on the sample set used differing numbers of factors were recommended. For comparative purposes, three factors were used per calibration (the average of the number recommended for all calibrations).

The standard error of cross validation (SECV) (determined from the residuals of each cross validation phase), the standard error of calibration (SEC) (determined from the residuals of the final calibration) and the co-efficient of determination ($R^2$) were used to assess calibration performance. The ratio of performance to deviation (RPD) (Williams and Sobering 1993), calculated as the ratio of the standard deviation of the reference data to the SECV, was also used to assess calibration performance. Determination of the RPD allows comparison of calibrations developed for different wood properties that have differing data ranges, the higher the RPD, the more accurate the data fitted by the calibration. Though an RPD of $>2.5$ is considered satisfactory for screening, it has been shown that calibrations with an RPD of approximately 1.5 can be useful for initial sample screening (Schimleck et al. 2003a).

Lignin and basic specific gravity calibrations were developed for each set of 40 NIR spectra. Calibration statistics ($R^2$, standard errors) were compared to determine what sampling methodology provided the strongest calibrations. Owing to the small size of the sample set the calibrations were not tested on a prediction set.

## Results

### Whole-tree chip calibrations

Calibrations obtained for the whole-tree chips (green, dried and milled after drying) are reported in Table 2. The calibrations for the whole-tree chips are reported first, as we expected these calibrations would give the best statistics as whole-tree chips best represent the whole-tree.

The milled chips gave a moderate $R^2$ (0.73) for insoluble lignin, while $R^2$ for whole-tree basic specific gravity (BSG), ranged from 0.67 (Chip BSG) to 0.70 (Disc BSG). Standard errors were relatively high for each property and RPD ranged from 1.26 to 1.57. An RPD of approximately 1.5 (Schimleck et al. 2003a) is considered a minimum for ranking trees hence the accuracy of the BSG calibrations are unsatisfactory for ranking purposes, while the insoluble lignin calibration (Fig. 1) may be suitable for initial screening.

The lignin calibration based on dry chips gave statistics that were similar to those obtained using milled chips, while the BSG calibrations were weaker with $R^2$ ranging from 0.45 to 0.51 and RPD ranging from 1.05 to 1.10. The calibrations based on green chips provided surprising results with the calibrations for the two

**Table 2** Wood property calibrations obtained using NIR spectra collected from whole-tree chips

| Wood property | Factors | $R^2$ | SEC | SECV | RPD |
|---|---|---|---|---|---|
| Milled chips | | | | | |
| Chip BSG | 3 | 0.67 | 0.02 | 0.03 | 1.26 |
| Disc BSG | 3 | 0.70 | 0.02 | 0.03 | 1.28 |
| Insoluble lignin | 3 | 0.73 | 0.43 | 0.52 | 1.57 |
| Dry chips | | | | | |
| Chip BSG | 3 | 0.45 | 0.03 | 0.03 | 1.05 |
| Disc BSG | 3 | 0.51 | 0.03 | 0.03 | 1.10 |
| Insoluble lignin | 3 | 0.70 | 0.45 | 0.59 | 1.38 |
| Green chips | | | | | |
| Chip BSG | 3 | 0.70 | 0.02 | 0.02 | 1.55 |
| Disc BSG | 3 | 0.74 | 0.02 | 0.02 | 1.66 |
| Insoluble lignin | 3 | 0.50 | 0.58 | 0.74 | 1.11 |

NIR spectra of the intact chips were obtained using a FOSS NIRSystems 6500, while spectra for the milled chips were obtained using a FOSS NIRSystems 5000

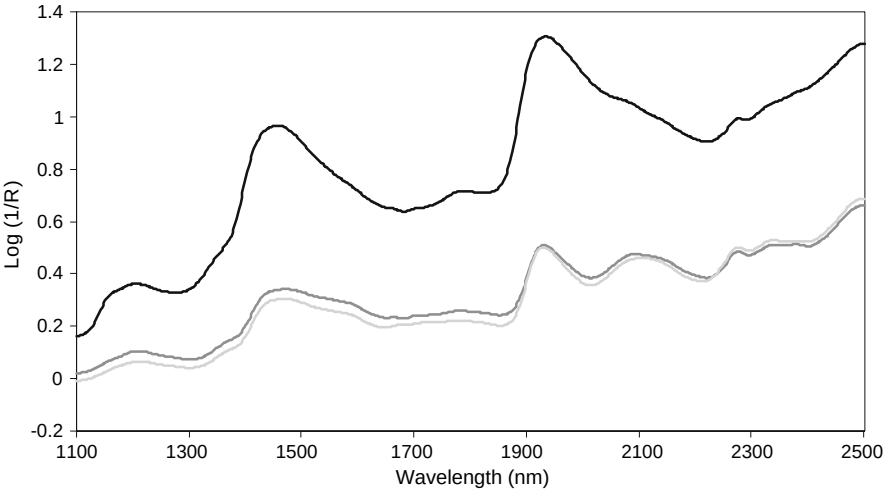


**Fig. 1** NIR diffuse reflectance spectra of the same sample when collected from green chips (*black line*), dry chips (*light grey line*) and milled chips (*grey line*)

measures of whole-tree BSG providing stronger statistics than those obtained for the dry chips and milled chips [$R^2 = 0.70$ (Chip BSG) and 0.74 (Disc BSG), and RPD = 1.55 (Chip BSG) and 1.66 (Disc BSG)]. The calibration for lignin based on green chips was not as successful as that based on milled dried chips with the $R^2$ and RPD falling to 0.50 and 1.11, respectively. We expected that calibrations based on green chips would have weaker statistics because several strong water absorption bands in the NIR spectrum (Fig. 2) obscure important spectral information.

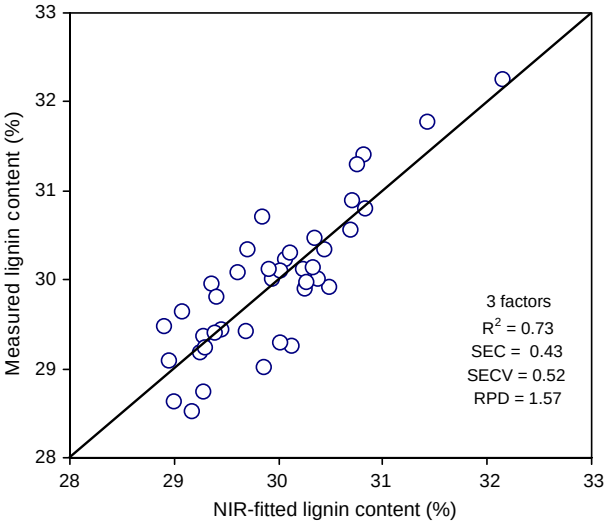


**Fig. 2** Relationship between laboratory-determined whole-tree insoluble lignin content and NIR-fitted whole-tree insoluble lignin content. NIR spectra were collected from milled chips

## Calibrations based on spectra collected from holes drilled at breast height (simulated standing trees) and milled increment cores

Calibrations for Chip BSG, Disc BSG and lignin based on NIR spectra collected from milled increment cores and NIR spectra collected from the radial surface of holes drilled into bolts are reported in Table 3.

The BSG calibrations based on milled 12 mm cores gave better statistics, in terms of RPD, than those obtained using milled whole-tree chips. In comparison, the calibration for lignin was noticeably weaker than what was obtained using milled chips.

**Table 3** Wood property calibrations obtained using NIR spectra collected from milled increment cores and holes drilled into bolts from breast height

| Wood property | Factors | $R^2$ | SEC | SECV | RPD |
|---|---|---|---|---|---|
| Milled BH core | | | | | |
| Chip BSG | 3 | 0.63 | 0.02 | 0.03 | 1.35 |
| Disc BSG | 3 | 0.63 | 0.02 | 0.03 | 1.38 |
| Insoluble lignin | 3 | 0.49 | 0.59 | 0.72 | 1.14 |
| BH hole | | | | | |
| Chip BSG | 3 | 0.45 | 0.03 | 0.03 | 1.09 |
| Disc BSG | 3 | 0.48 | 0.03 | 0.03 | 1.14 |
| Insoluble lignin | 3 | 0.21 | 0.73 | 0.91 | 0.90 |

NIR spectra of the milled cores were obtained using a FOSS NIRSystems 5000, while spectra from the drilled holes were obtained using an Ocean Optics fiber-optic probe system

Calibrations based on NIR spectra collected from the surface of the hole were weaker than those developed for the milled chips with $R^2$, for both lignin and basic specific gravity <0.50.

## Calibrations based on intact cores

Rather than going to the trouble of milling cores prior to NIR analysis, it is possible that calibrations based on NIR spectra collected from intact cores will provide comparable statistics to those based on milled samples. Calibrations based on NIR spectra collected from sections of radial strips indicate that excellent calibration statistics can be obtained for properties, such as density, microfibril angle and stiffness (Jones et al. 2005). While the statistics were strong, it should be noted that NIR spectra were collected from a small region of wood (10 mm wide) for which the wood properties were accurately known. In comparison, in this study, we attempted to correlate spectra collected from the surface of the core with whole-tree properties.

Calibrations obtained using NIR spectra obtained from intact 12 mm cores using the FOSS NIRSystems 5000 are reported in Table 4. The calibrations were based on NIR spectra collected in 10 mm increments from the rough surface of the cores while green and dry, and from a smooth core surface (bandsaw cut) also, when green and dry.

**Table 4** Wood property calibrations obtained using NIR spectra collected from intact 12 mm increment cores using the FOSS NIRSystems 5000

| Wood property | Factors | $R^2$ | SEC | SECV | RPD |
|---|---|---|---|---|---|
| BH green core rough | | | | | |
| Chip BSG | 3 | 0.33 | 0.03 | 0.04 | 0.96 |
| Disc BSG | 3 | 0.38 | 0.03 | 0.04 | 0.89 |
| Insoluble lignin | 3 | 0.29 | 0.69 | 0.98 | 0.84 |
| BH green core smooth | | | | | |
| Chip BSG | 3 | 0.48 | 0.03 | 0.04 | 1.03 |
| Disc BSG | 3 | 0.42 | 0.03 | 0.04 | 1.04 |
| Insoluble lignin | 3 | 0.20 | 0.74 | 1.12 | 0.73 |
| BH dry core rough | | | | | |
| Chip BSG | 3 | 0.35 | 0.03 | 0.04 | 1.00 |
| Disc BSG | 3 | 0.41 | 0.03 | 0.04 | 1.02 |
| Insoluble lignin | 3 | 0.33 | 0.67 | 0.85 | 0.96 |
| BH dry core smooth | | | | | |
| Chip BSG | 3 | 0.55 | 0.02 | 0.03 | 1.13 |
| Disc BSG | 3 | 0.50 | 0.03 | 0.03 | 1.10 |
| Insoluble lignin | 3 | 0.29 | 0.69 | 0.93 | 0.88 |

The calibrations are based on NIR spectra collected from the rough and smooth (cut) surface of the cores while green and dry

Calibrations based on spectra collected from green cores gave statistics that were weaker than those obtained using milled chips regardless of the surface (rough or smooth) examined. The strongest calibrations (whole-tree BSG) were obtained using spectra collected from the smooth surface of dry cores.

Calibrations obtained using NIR spectra collected from intact 12 mm cores using the Ocean Optics fiber-optic probe system are reported in Table 5.

Calibrations obtained using the Ocean Optics fiber-optic probe system provided statistics that were noticeably weaker than what was obtained using milled chips. Generally, $R^2$ were <0.50 and RPDs were approximately one or less. The highest $R^2$ (0.52) was obtained for the Chip BSG calibration obtained using spectra collected from the smooth surface of green cores.

## Calibrations based on drill shavings

While taking a core from a tree is considered nondestructive, the hole left in a tree after the extraction of a 12 mm core is still quite large and if the tree is small then the hole can pose a threat to the structural integrity of the tree. With this in mind, the option of using shavings from drilling a 6.25 mm diameter hole into the sampled trees was investigated. Calibrations based on drill shavings when green, dried, and dried and milled are given in Table 6.

**Table 5** Wood property calibrations obtained using NIR spectra collected from intact 12 mm increment cores using the Ocean Optic fiber-optic probe system

| Wood property | Factors | $R^2$ | SEC | SECV | RPD |
|---|---|---|---|---|---|
| BH green core rough | | | | | |
| Chip BSG | 3 | 0.40 | 0.03 | 0.04 | 1.00 |
| Disc BSG | 3 | 0.40 | 0.03 | 0.04 | 1.00 |
| Insoluble lignin | 3 | 0.20 | 0.74 | 0.92 | 0.90 |
| BH green core smooth | | | | | |
| Chip BSG | 3 | 0.52 | 0.03 | 0.04 | 0.96 |
| Disc BSG | 3 | 0.47 | 0.03 | 0.04 | 0.99 |
| Insoluble lignin | 3 | 0.25 | 0.71 | 0.94 | 0.87 |
| BH dry core rough | | | | | |
| Chip BSG | 3 | 0.45 | 0.03 | 0.04 | 0.86 |
| Disc BSG | 3 | 0.44 | 0.03 | 0.05 | 0.82 |
| Insoluble lignin | 3 | 0.23 | 0.72 | 1.04 | 0.79 |
| BH dry core smooth | | | | | |
| Chip BSG | 3 | 0.39 | 0.03 | 0.03 | 1.05 |
| Disc BSG | 3 | 0.41 | 0.03 | 0.03 | 1.07 |
| Insoluble lignin | 3 | 0.33 | 0.67 | 0.95 | 0.86 |

The calibrations are based on NIR spectra collected from the rough and smooth (cut) surface of the cores while green and dry

**Table 6** Wood property calibrations obtained using NIR spectra collected from intact drill shavings when the samples were green, dry and milled

| Wood property | Factors | $R^2$ | SEC | SECV | RPD |
|---|---|---|---|---|---|
| BH shavings green | | | | | |
| Chip BSG | 3 | 0.58 | 0.02 | 0.03 | 1.13 |
| Disc BSG | 3 | 0.54 | 0.03 | 0.03 | 1.12 |
| Insoluble lignin | 3 | 0.44 | 0.61 | 0.82 | 1.01 |
| BH shavings dry | | | | | |
| Chip BSG | 3 | 0.35 | 0.03 | 0.04 | 0.90 |
| Disc BSG | 3 | 0.33 | 0.03 | 0.04 | 0.85 |
| Insoluble lignin | 3 | 0.44 | 0.62 | 0.75 | 1.10 |
| BH shavings dry, milled | | | | | |
| Chip BSG | 3 | 0.28 | 0.03 | 0.04 | 0.89 |
| Disc BSG | 3 | 0.34 | 0.03 | 0.04 | 0.90 |
| Insoluble lignin | 3 | 0.58 | 0.53 | 0.70 | 1.17 |

Spectra were obtained using a FOSS NIRSystems 5000

Weak calibrations were obtained using sawdust from holes drilled at breast height. Of the different sets, green sawdust from breast height gave the strongest $R^2$ but the RPDs were low.

## Discussion

The performance of wood property calibrations obtained using NIR spectra collected from samples obtained using a variety of different sampling options was investigated. The strongest calibrations were obtained using NIR spectra from intact chips with NIR spectra from intact green chips providing the strongest calibrations for BSG. The green chip calibrations also provided the highest RPD, which was significant as the RPD indicated that the Disc and Chip BSG calibrations would be suitable for ranking trees. When compared to the results obtained using dried chips, the stronger statistics obtained for green chips were unexpected as it was thought that calibration performance would suffer owing to the presence of several strong absorption bands in the NIR spectrum caused by water (Blosser 1989). The significant absorption of NIR radiation is caused by the hydrogen bonds in water and result in broad peaks that obscure spectral information derived from other compounds (Abrams et al. 1988). Blosser (1989) noted that the presence of the moisture bands limits the usefulness of NIR spectroscopy for high-moisture feedstuffs, but also reported that NIR spectroscopy had been used successfully with a wide variety of high-moisture materials inspite of the strong absorption bands caused by water. Past studies based on green wood (Meder et al. 2003; Schimleck et al. 2003b; Thygesen 1994) have shown that it is possible to use NIR spectroscopy to estimate physical wood properties (density, stiffness), however, the errors

associated with the predicted properties were larger than those obtained for dried wood.

Calibrations for BSG based on milled increment cores were similar to those based on milled chips, while for lignin, the results for milled cores were noticeably weaker than those for milled chips. The results for BSG indicate that little would be lost, in terms of calibration accuracy, if milled cores were used to estimate whole-tree BSG. The results for BSG support the findings of other studies (Schimleck et al. 2005a, b, 2006) that have investigated the development of wood property calibrations using core NIR spectra and whole-tree wood property data. The calibration statistics reported in this study for lignin are weaker than those found by Schimleck et al. (2006) for several eucalypt species and hybrids grown in Brazil, where $R^2$ for lignin ranged from 0.77 to 0.79 and $RPD_c$ ranged from 1.77 to 1.90. Calibration statistics reported by Schimleck et al. (2006) for BSG ($R^2$ ranged from 0.64 to 0.76 and $RPD_c$ ranged from 1.50 to 1.93) were similar to those reported for BSG in this study. In a study based on hybrid poplar, Schimleck et al. (2005a) reported calibrations for whole-tree cellulose content and pulp yield based on whole-tree chip and increment core NIR spectra that had much stronger $R^2$ (0.89 to 0.96).

Wood property calibrations were also developed using NIR spectra collected from the surface of intact cores using a FOSS NIRSystems 5000 spectrometer and an Ocean Optics spectrometer fitted with a fiber-optic probe. NIR spectra were collected from the rough surface of the cores while green and dry and from a smooth surface (cut with a bandsaw) also when green and dry. The calibrations based on spectra obtained using dry cores with a smooth surface gave the strongest calibrations for BSG [$R^2 = 0.50$ (Disc BSG) and 0.55 (Chip BSG)] but statistics were far weaker than those obtained using green chips or milled chips. Calibrations based on NIR spectra collected from green cores were weak regardless of the surface or spectrometer used. The results obtained using NIR spectra collected from intact cores were weaker than expected based on calibrations reported by Schimleck et al. (2003b), who found that moisture content and surface roughness had a negative influence on calibration statistics but not to the degree observed here. It should be noted that Schimleck et al. (2003b) estimated properties of sections of radial strips not whole-tree properties. The primary advantage of using intact cores for spectroscopy is that it avoids milling the core and presumably saves time, however, we found that it was extremely time-consuming to collect all the required spectra, for example, a core 100-mm long required ten spectra to be collected using the 10-mm wide window. In an attempt to save time, the number of spectra collected per core could be reduced, for example, three spectra could be collected, one near the pith, another near the middle of the core and the third near the bark, and then averaged, but if calibration statistics remain poor then there may be little point in pursuing this approach.

NIR spectra were also collected using a fiber-optic probe inserted in a hole drilled into a bolt removed at breast height. The main advantage of using this approach is that it provides real-time assessment of wood properties. However, in practice there are several disadvantages, including resin bleeding into the core hole, resin adhering to the probe (in addition, the resin is difficult to remove), the difficulty of designing

a suitable probe for NIR analysis and the performance of fiber-optic probe systems compared to bench-top NIR spectrometers. NIR probes are designed with a window at their end, and when obtaining an NIR spectrum, NIR energy passes through the window and is absorbed and reflected by the surface in front of it, and the reflected energy is collected to give a spectrum. For a hole drilled into a tree, this approach will only provide a spectrum from the wood at the end of the hole, while a spectrum is desired from the radial surface of the hole. In this study, we used a short NIR probe (length 12.5 mm) positioned at an angle of 45° to collect spectra. While the spectra were adequate for analysis, we found that the length of the probe coupled with the fiber-optic cable required a hole of at least 25 mm in diameter, which is larger than the hole obtained if an increment core is removed. Other options for collecting spectra from core holes include a probe fitted with a prism angled at 45° to deflect the NIR beam onto the radial surface or a fiber-optic bundle without a barrel fitted at the end so that it retains some flexibility and can therefore be bent to facilitate the collection of spectra from the radial surface of the core hole. The poor calibration statistics obtained using NIR spectra collected using a fiber-optic probe inserted into a tree in this study indicates that calibrations will not be sufficiently accurate for practical purposes. However, further investigation is required of the various fiber-optic options and NIR instruments for collecting NIR spectra from trees to fully assess the utility of this approach.

The final approach investigated involved collecting NIR spectra from shavings obtained when a 6.25-mm diameter hole was drilled into each of the 40 trees. This approach is attractive as it is far less time-consuming and labor intensive than coring trees and it leaves a small hole compared to the hole obtained when a 12 mm increment core is removed, which is particularly important for small trees. In addition, the shavings can be quickly dried and easily milled. Unfortunately, the calibrations based on shavings (green or dry) were weak compared to those obtained for the chips and milled cores. We expected the results for shavings to be similar to those of milled cores, and recommend that the use of milled shavings for NIR analysis be further investigated using larger and more variable data sets.

## Conclusion

Calibrations for whole-tree lignin and BSG based on NIR spectra from whole-tree chips (milled or intact) had the strongest statistics. Calibrations obtained using NIR spectra obtained from milled increment cores had similar statistics. Calibrations based on NIR spectra obtained from intact cores provided weaker statistics than those obtained using milled cores. For intact cores, sample preparation was minimal, however, collecting spectra from the cores in 5 or 10 mm increments was far more time consuming than drying and milling the cores and collecting spectra from the ground wood. Other options for sampling the tree (drill shavings, collecting spectra from tree in the field, etc.) gave errors that were too large for practical purposes. If an increment core is going to be used to estimate whole-tree properties, it is recommended that it be dried and milled prior to analysis.

Increasingly, NIR spectroscopy is being investigated as a solution for problems were data are required real-time or where large numbers of samples must be analyzed quickly. In terms of real-time analysis the application of NIR spectroscopy has been explored both in the forest and in manufacturing environments. The results from this study demonstrate that there are several practical difficulties inherent in trying to measure the wood properties of *P. taeda* in the field, however, some of these problems (for example resin bleeding into core holes) are non-issues for other important plantation species, such as eucalypts, and improvements in the design of fiber-optic probes should improve results. Several studies have also investigated the utilization of NIR spectroscopy for measuring the properties of crops as they are being harvested (Haeusler et al. 2002; Paul and Pfitzner 2004; Sinnaeve et al. 2004) and similar efforts have commenced in forestry where the wood properties of chainsaw chips produced when a tree or log is cut have been examined using NIR (Acuna and Murphy 2006). Initial results are promising and research in this area will continue. NIR spectroscopy has also been assessed for on-line assessment of wood properties both as the wood is entering the manufacturing facility (Axrup et al. 2000) and also potentially while the product is being manufactured (Meder et al. 2002, 2003; Rials et al. 2002). The ultimate aims of such work are a reduction in process variability and a subsequent improvement in product quality, and both should be achieved assuming that the applications that NIR spectroscopy is being used for are appropriate; again research in this area will continue.

NIR spectroscopy is also becoming an important tool for tree breeders in assessing the wood properties of plantation grown trees (Schimleck 2007). Ideally wood properties, either on a whole-tree basis or radially (to give the equivalent of a core average), would be assessed in the field but presently analysis is limited to the laboratory. The most time consuming aspect of such work is the NIR analysis of sections of radial strips (cut from increment cores) for the examination of wood property variation radially or for the determination of core averages. NIR analysis of radial strips is also limited to relatively low-spatial resolutions at present, with none of the instruments currently available able to accurately estimate wood properties at a spatial resolution of 2 mm (Jones et al. 2007). Hence, analysis would greatly benefit from the availability of a high-energy NIR spectrometer that can collect spectra rapidly, and at high-spatial resolution.

While the advantages of NIR spectroscopy for wood property determination are numerous, the uptake of the technology in the forestry sector has been slow. Various reasons for the slow uptake exist and that include the cost and time involved in establishing new calibrations, the doubts about the transferability of calibrations to samples from new locations, the ongoing need to update calibrations, the applicability of NIR to samples whose moisture content varies greatly and the unrealistic expectations on behalf of new users.

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